

What is claimed is:

1. A process for producing an ammonium polythiomolybdate of the formula  $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$  where  $n$  is 0, 1 or 2 comprising:
  - (a) reacting an aqueous ammoniacal molybdate solution with hydrogen sulfide gas at superatmospheric pressure until the  $\text{H}_2\text{S}$  is no longer absorbed by the solution, said solution and said gas being in a closed system and the flow of said gas being regulated at an elevated pressure to form a slurry consisting essentially of a solid essentially all of which is ammonium tetrathiomolybdate containing a portion of the starting molybdenum and a mother liquor containing the balance of the molybdenum;
  - (b) heat soaking the reaction product of step (a) at elevated temperatures up to about  $200^\circ\text{C}$  in a closed reactor in the presence of elemental sulfur at a pressure of 600-1000 psig whereby the ammonium tetrathiomolybdate is converted to  $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$ ;
  - (c) cooling said slurry to ambient temperature;
  - (d) separating said solid from the major portion of said mother liquor;
  - (e) washing said solid with water followed by removing the resulting water washes to remove the remaining portion of said mother liquor and soluble impurities from said solid; and
  - (f) drying the resulting washed solid at ambient temperature to form the  $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13} \bullet n \text{H}_2\text{O}$ .
2. The process of claim 1 wherein the ammonium polythiomolybdate is  $(\text{NH}_4)_2\text{Mo}_3\text{S}_{13}$ .

3. The process of claim 1 wherein the ammoniacal solution comprises MoO<sub>3</sub>, (NH<sub>4</sub>)<sub>2</sub>S and elemental sulfur.
4. The process of claim 1 wherein the pressure in step (a) is 5-50 psig.
5. The process of claim 1 wherein the temperature in step (b) is 175-200°C.
6. The product prepared by the process of claim 1.